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PHYSICAL EVALUATION OF 87/10.5/2.5 BA/2-EHA/MAD ELASTOMER

Report by

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A B S T R A C T

The 87/10.5/2.5 BA/2-EHA/MAD elastomer was evaluated for physical properties. The properties possessed by this elastomer may be adequate for heart valve application. Additionally, the excellent low temperature stiffness in flexure of this elastomer appears advantageous as a substrate material in the dilaminar acrylate glove.

Physical Evaluation of 87/10.5/2.5 BA/2-EHA/MAA Elastomer

I. INTRODUCTION

Technical Report No. 6118 entitled "Vascular Prostheses from Foamed Acrylate-Amide Elastomer", by Carl A. Nielson, described the preparation and clinical evaluation of the acrylate-amide terpolymer elastomers in vascular prostheses. In addition, this material has shown some promise as a possible tracheal prostheses as described in T. R. No. 5952 entitled "Acrylate-Amide Tracheal Prostheses."

Since the Standard 10 acrylate-amide elastomeric material has consistently shown good tissue response, it was thought that a more flexible acrylate material could be of possible use as a material for fabrication of heart valve leaflets.

This report describes the preparation and properties of an elastomer composed of 87/10.5/2.5 parts by weight of butyl acrylate/ 2 ethyl hexyl acrylate/methacrylamide.

A. Experimental

The basic recipe used is as follows:

Ingredient	Parts	Supplier
Butyl Acrylate	87	Rohm & Haas Co. W. Wash. Sq. Philadelphia, Pa.
2-Ethyl Hexyl Acrylate	10.5	" " "
Methacrylamide	2.5	" " "
Santomerse SX	2.5	Monsanto Chem. Co. St. Louis, Ill.
Water (Deionized)	155	" " "
Sulfuric Acid	10 mls 10%	Reagent Grade
Potassium Chloride	0.344	" "
Sodium Thiosulfate	0.04	" "
Potassium Persulfate	0.04	" "

B. Polymerization

The polymerization was run in a 5 liter reaction flask. The equipment used and the procedure followed is essentially described in T. R. No. 5701 entitled, "Five Liter Polymerization Reactor." Figure 1 is a graphical illustration of the conversion of monomers to polymer attaining 94% conversion in 24 hours. The new terpolymer latex possessed a final pH of 2.2, residual monomer of 1.2% and a viscosity of 9 cps. at 60 R.P.M. using the Brookfield Viscometer.

C. Compounding

Compounding of the new acrylate terpolymer was accomplished as follows:

Material	Parts
87/10.5/2.5 BA/2-EHA/MAD Elastomer	100
Rohm & Haas Mc 387 Polyethylmethacrylate	37
Formaldehyde (added as Formalin Solution)	1.765

The ingredients were thoroughly mixed with a flat paddle type stirrer for 10 minutes and strained through gauze into a suitable container.

D. Film Casting

1A) One series of test films was obtained from the compounded latex as follows:

a) The films were coagulative dip cast using a 25% solution of calcium nitrate in ethanol as a coagulant over aluminum forms for 5 minutes so that a 30-35 mil thickness was obtained.

b) Films were removed from the form and washed in di-ionized water for at least one hour.

c) The films were dried overnite at room temperature and then dried at 50°C. for 2 hours in a convection type oven.

c) A final curing of the films by one hour at 100°C. in a circulating air oven.

1B) Half of the cured films were further treated as follows:

a) One hour in boiling 5% dilute formaldehyde

b) Rinsed and patted dry

c) One hour in a steam autoclave at 258°F. (125°C.) and 18 lbs. of steam pressure

2A). Another series of films was obtained as in 1A from compounded latex which had been heat aged for 3 hours at 100°C. in a water bath.

2B) Half of the films from 2A were treated as described in 1B.

E. Physical Testing

The four series of test films were evaluated for the following physical properties; stress-strain curve at 20"/min, tear resistance, stiffness in flexure, tensile fatigue, per cent stain resistance, swelling index and per cent solubility.

III. RESULTS AND DISCUSSION

All the physical data obtained are summarized in Table I. Stress-strain curves of the various films are graphically illustrated in Fig. 2. From this graph it can be seen that the stress-strain curves were shifted toward the stress axis when the films were both post-treated in boiling dilute formaldehyde and autoclaved and when the films were obtained from compounded latex which had been heat aged for 3 hours. These results are quite comparable to those obtained with the Standard 90/7.5/2.5 BA/MMA/MAD elastomer.

The curing procedure for internal body prostheses as reported in T.R. No. 6118 consists of heat aging the compounded latex for 3 hours at 100°C., curing the resultant films by boiling in dilute formaldehyde for one hour followed by a final cure in an autoclave at 125°C. plus 18 lbs. of steam pressure. Curve 2B represents physical properties obtained on material so treated. With a tensile strength of 1090 p.s.i. an elongation of 393%, a tear strength of 106 p.i., and a tensile fatigue at 80% of breaking load of 155 cycles, this material possesses a number of physical properties which may be adequate for heart valve use.

Figure 3 is a graphical illustration of the low temperature stiffness in flexure properties of this material compared to that of a standard acrylate-amide elastomer, e.g., a 90/7.5/2.5 BA/MMA/MAD standard compounded material. The 2B material is approximately 8 times better than the standard acrylate-amide material at -40°C. However, at +10°C. both materials possess approximately the same flexural stiffness. In a further effort to attain more flexibility the 87/10.5/2.5 BA/2-EHA/MAD latex was cast as is, e. g. without reinforcing filler. Unfortunately, the resultant film obtained was much too tacky for use. However, as good low temperature properties are required for the dilaminar acrylate glove, this new elastomer could be a possible replacement for the standard acrylate-amide elastomer as the substrate material. Accordingly, a dilaminar film was cast which consisted of a 30 mil substrate of 87/10.5/2.5 BA/2-EHA/MAD compounded elastomer and a 4 mil outer layer of 73/27 BA/AN elastomer.

This test film was dried and cured in the usual manner and physical properties obtained are listed in Table II.

Referring to the low temperature properties listed in Table II at -40°C. the flexural stress obtained is lower than that of a comparable acrylate-amide elastomer. However, at higher temperatures the stresses obtained are slightly higher than that of the standard elastomer. All other properties with the exception of stain resistance were equal to or slightly better than that of the standard elastomer. The new material was approximately 9 times more resistant to staining than the standard elastomer.

IV. CONCLUSIONS AND RECOMMENDATIONS

The 87/10.5/2.5 BA/2-EHA/MAD elastomeric material possesses physical properties which may be adequate for heart valve utilization. In addition, the excellent low temperature properties of this elastomer is indicative of possible use as the substrate material in the dilaminar acrylate glove.

Therefore the author recommends that:

1) Work should continue with formulations related to the above material in an effort to attain greater flexibility.

2) A dilaminar glove should be fabricated using the new elastomer as the substrate material and field tested.

Acknowledgement

The author wishes to acknowledge the work done by Mr. John Hodge for his efforts in the physical testing of the elastomeric materials.

TABLE I

Modulus At 100	200	300	400	500	600	%E	Tensile Strength	Tear Resis.	Tensile Fatigue	Low Temp. Flexural -40°C	-30°C	-20°C	-10°C
156	223	307	423	623	926	700	1400	89	44	1500	800	650	550
415	620	845	1070			445	1185	135					
236	426	640	850	1100		530	1225	96	106	1700	1200	1100	850
326	556	833				393	1090	106	155	2200	1700	1400	1200

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I ar sis.	Tensile Fatigue	Low Temp. Flexural -40°C	-30°C	-20°C	-10°C	0°C	+10°C	S.R. %	Swell Index	% Sol
9	44	1500	800	650	550	450	350	17.9	19.1	29.3
5									9.95	28.8
6	106	1700	1200	1100	850	730	650	7.0	6.46	21.5
6	155	2200	1700	1400	1200	800	780	7.0	7.5	27.2

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TABLE II

Film	Bilaminar Acrylate Film
Stress at:	
50%	143
100%	216
200%	353
300%	533
400%	916
% Elongation	433
Tensile Strength	1243
Tear Resistance	101
Tensile Fatigue	26
% Stain Resistance	61
Low Temperature Flexural:	
-40°C	12,000
-30°C	9,000
-20°C	3,900
-10°C	2,900
0°C	2,300
+10°C	1,450
Swelling Index	117
% Solubility	28.2

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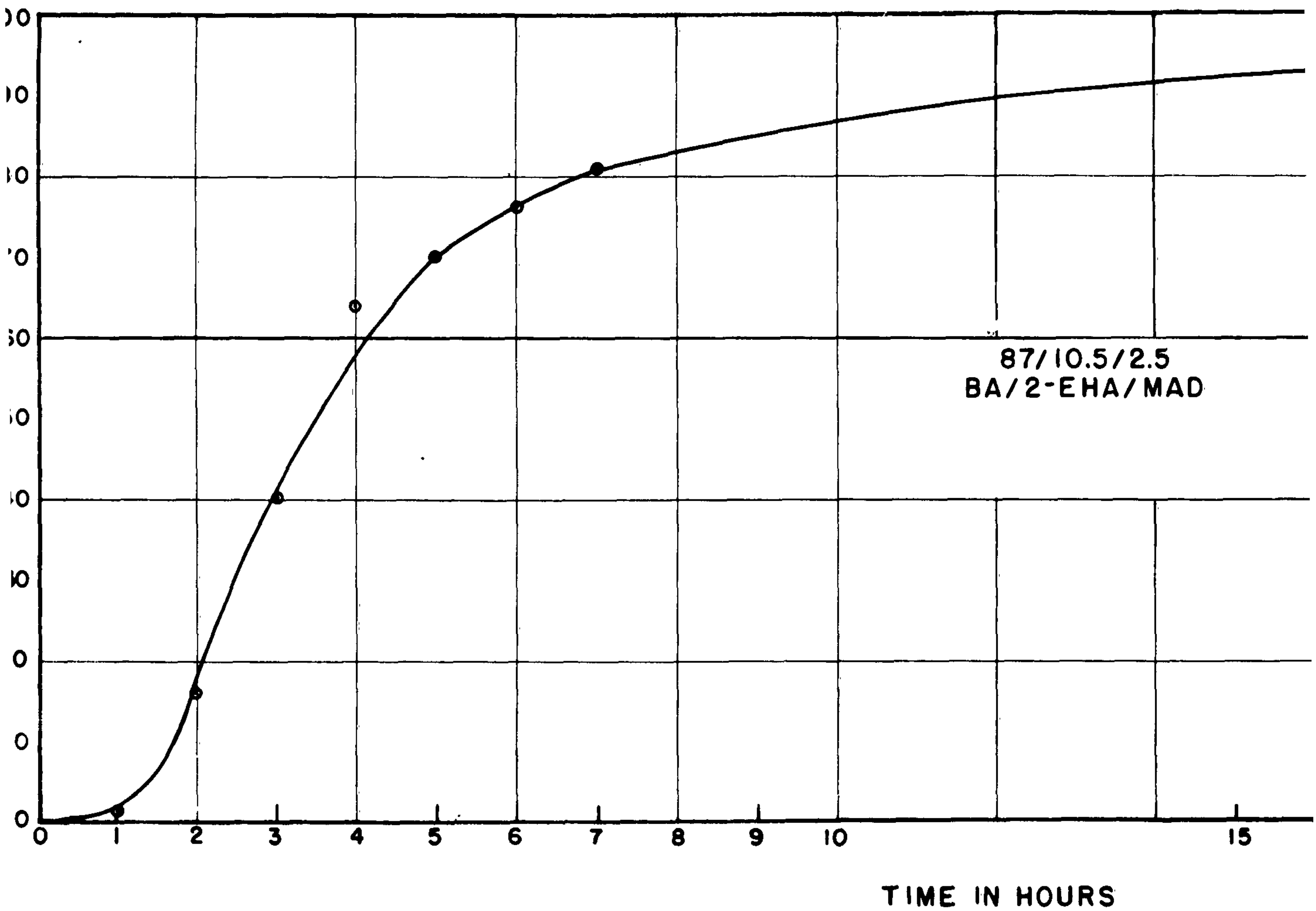
Fig.I

POLYMERIZATION CONVERSION CUR



2

Fig.1

ERIZATION CONVERSION CURVE

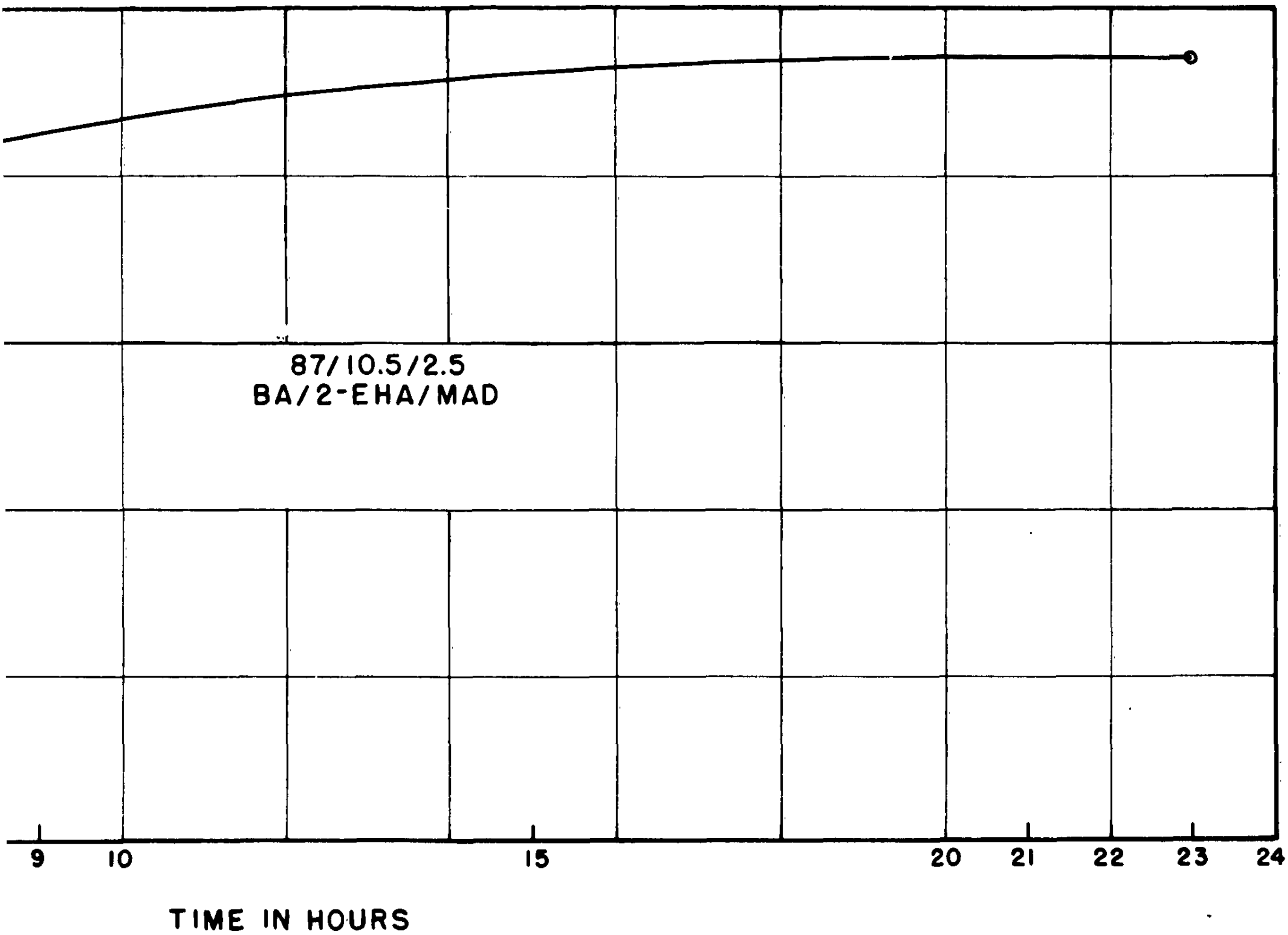


Fig. 2

LOW TEMPERATURE-STIFFNESS IN FLEXURE

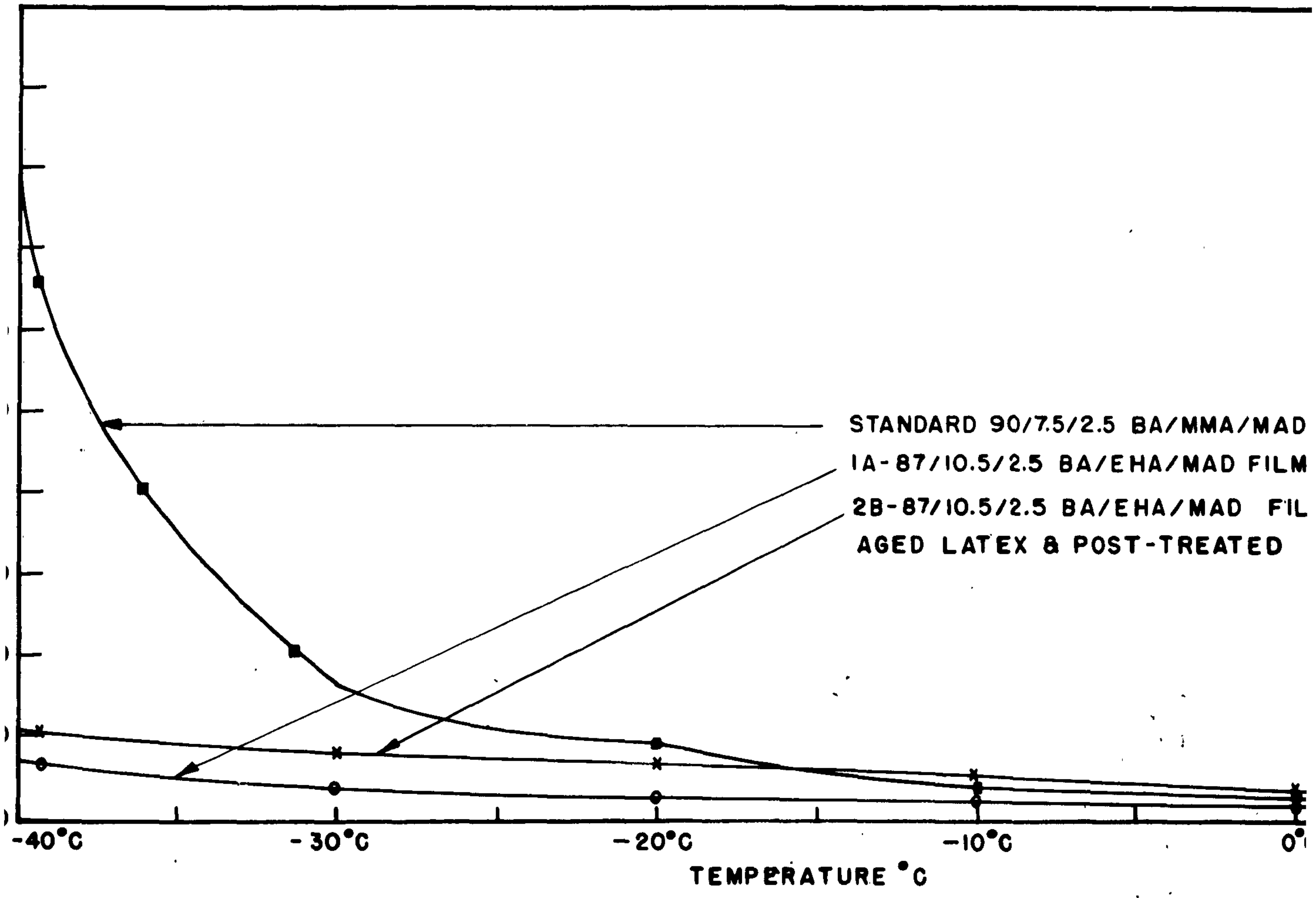


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Fig.2

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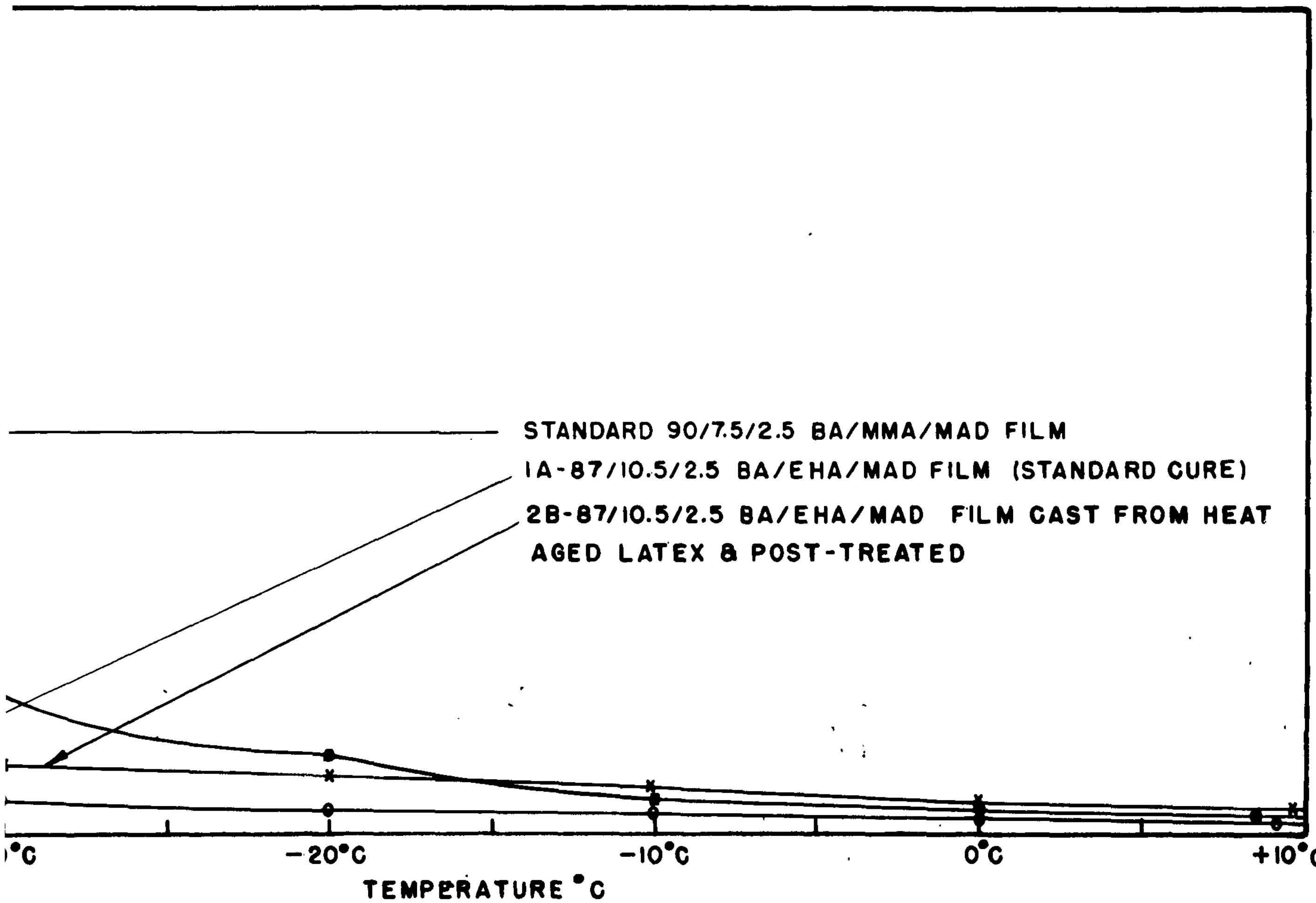
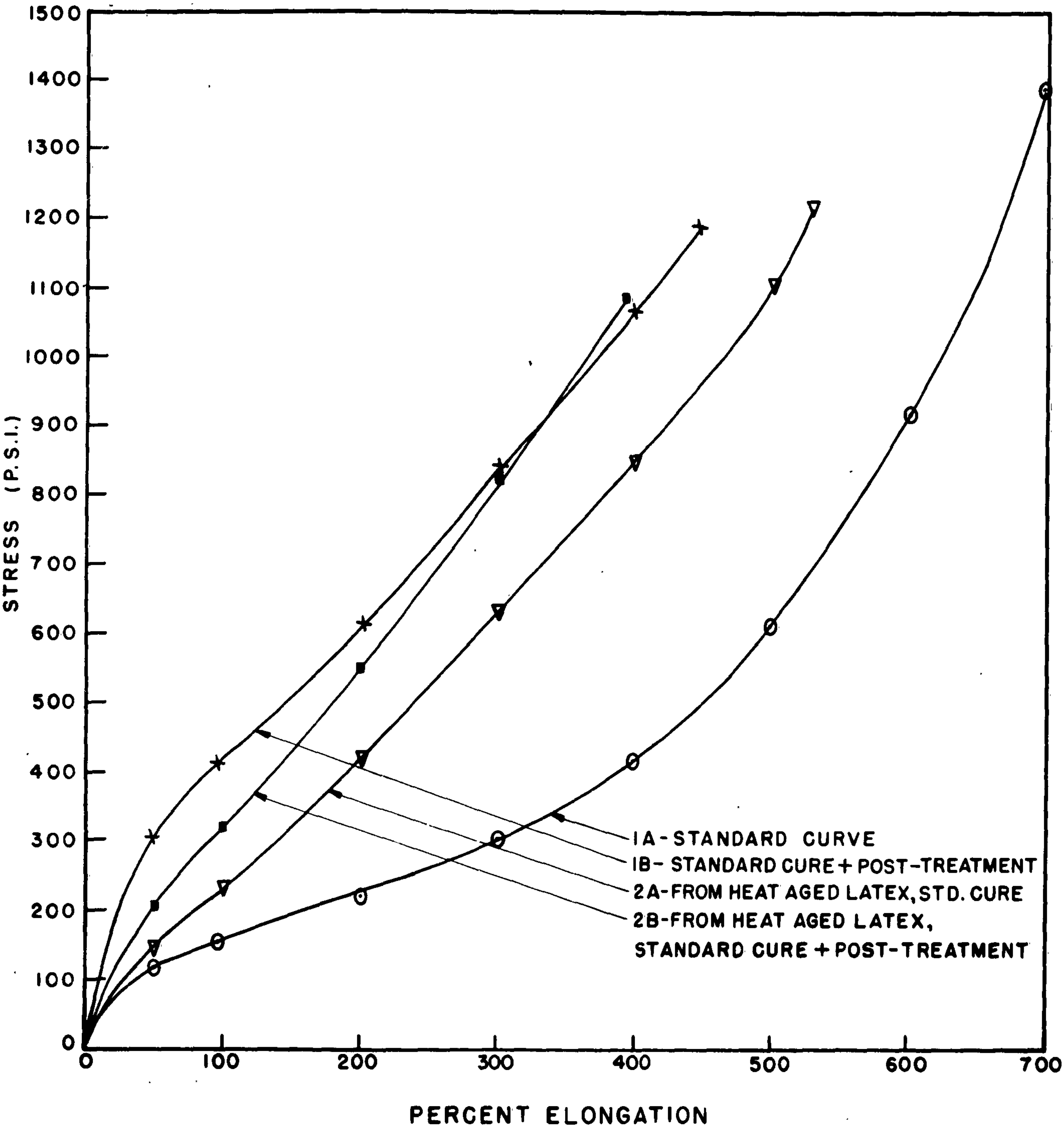


Fig. 3

STRESS-STRAIN CURVE



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1. Internal body Mat'l
2. Elastomer material
3.
4.

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WRAMC FORM 0183 (ONE TIME)
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